

# Single and Binary Adsorption of Reactive Blue and Red Dyes Onto Activated Carbon

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**Abstract** – This study aims to evaluate the ability of activated carbon to remove reactive blue and red dyes from aqueous solutions. Batch experiments were conducted to obtain equilibrium and kinetic data, as well as, optimum conditions such as: pH; temperature; contact time, and initial dye concentration. The results showed that the activated carbon can be successfully used in removing two dyes from wastewater and the removal efficiency up to 85% can be achieved when using activated carbon as reactive blue and red dyes adsorbent. FTIR spectroscopy analysis showed that the hydroxyl and carboxyl groups are the major groups that responsible for the adsorption process of two dyes. The kinetics data of two dyes fitted well the pseudo-second-order kinetic model. For both single and binary systems the experimental data fitted well to Langmuir isotherm model and the maximum uptake in single system was found to be 2.89 and 2.15 mg/g for reactive red and blue dyes, respectively. These values were reduced in binary system due to the competition between dyes molecules for binding sites onto activated carbon.

**Keywords** – Dyes, Adsorption, Activated Carbon, Equilibrium, Kinetics.

## I. INTRODUCTION

The pollution of environment by organic and/or inorganic pollutants has become one of the most serious environmental problems today and still requires the development of quick and simple methods for the removal of these pollutants. This is because they are highly toxic, non-biodegradable, and tend to accumulate causing different health problems in living organisms [1].

Disposal of effluents from the dyeing industry pose one of the major problem, because such effluents contain a number of pollutants including acids or bases, dissolved solids, surfactants, and dyes. Out of these, dyes are the most easily recognized contaminant because they are visible to the human eye. These dyes can cause harmful actions to various forms of aquatic-life. Dyestuff effluent is resistant to light and when discharged into the water bodies, it prevents the sunlight from penetrating through and reduces the aesthetic quality of water [2]. Anazo dyes are by far the most important class, accounting for over 50% of all commercial dyes, and having been studied more than any other class. A majority of the used dyes are azo reactive dyes which are bright in color due to presence of

one or several azobonds (-N=N-) associated with substituted aromatic structures[3, 4]. The most important characteristic of reactive dyes is formed covalent bonds with the substrate that is to be colored during the application process. Thus, the dye molecule contains specific functional groups that can undergo addition or substitution reactions with the OH<sup>-</sup>, SO<sub>3</sub>, and NH<sub>2</sub> groups present in fiber[5]. Most reactive dyes fall in the category of azo dyes. These dyes or their breakdown products are toxic to living organisms [6]. Dyes in wastewater are difficult to remove by conventional methods because they are stable to light, heat and oxidizing agents, as well as, they are not easily degradable [7].

Adsorption techniques are generally considered as the preferred means for removing organic substances due to their efficiency to separate chemical compounds from wastewater. The most widely used adsorbent for this purpose is activated carbon [8]. Furthermore, adsorption is an important step in industrial downstream processing. Adsorption process is often used at the end a treatment sequence for pollution control due to the high degree of purification that can be achieved. Activated carbon is being the most common adsorbent due to its effectiveness and versatility. This adsorbent is widely used in the chemical process industries and is playing important role in cleaning up plant effluent and municipal wastewater [9]. This study was undertaken to address the problems associated single and binary adsorption from aqueous solutions. This is essential for accurate design of adsorption systems as the effect of multicomponent interactions in the process effluent may cause deterioration in the adsorption capacity of activated carbon for dyestuffs.

The main objectives of this work are: (i) to characterize the physiochemical properties of activated carbon such as specific surface area and functional groups. (ii) to investigate the effects of manipulation of some experimental parameters e.g. pH, shaking time, adsorbent dose, initial concentration and temperature on the adsorption capacity of reactive blue H3R and red 3B dyes. (iii) to study a single and binary batch adsorption process. (iv) to investigate the isotherm model that can be used to describe the adsorption process. (v) to study the kinetics adsorption of these two dyes.

## II. MATERIALS AND METHODS

### II.I Materials

Granular activated carbon (AC) of particle size (0.5-0.75) mm, surface area of 911.581 m<sup>2</sup>/g, and bulk density of 711.8 kg/m<sup>3</sup> was used as adsorbent in this study. The activated carbon was washed with distilled water prior drying in electrical oven at 100 °C for 6 h.

Simulated stock solutions of reactive blue (RB) and reactive red (RR) dyes had been prepared by dissolving 1 g of each dye in one liter distilled water then diluted to the required concentration and kept in glass container at the room temperature. All chemicals used in this work were analytical reagent grade and were used without further purification. Table (1) shows the main properties of these dyes.

Table 1: Main properties of the dyes used in this study

Item	Reactive blue (RB)	Reactive Red (RR)
Trade name	Blue H3R	Red 3B
Origin	India	China
Status	Solid Powder	Solid Powder
Wave length (nm)	585	540
Solubility (g/l)	90	100
pH	6.2-6.5	6.2-6.5

### II.II Methods

#### II.II.I Fourier transformation infrared (FTIR) spectroscopy analysis

Various authors have used FTIR spectroscopy to detect vibration frequency changes in AC before and after adsorption. FTIR offers excellent information on the nature of the bands present on the surface of the adsorbent and also presents three main advantages as an analytical technique: it is fast, nondestructive and requires only small sample quantities [10]. FTIR technique was used in this study to examine the surface groups of the adsorbent and to identify those groups responsible for dye adsorption.

Two samples were prepared for FTIR analyses. The first sample contains 1 g of dried AC and represents the adsorbent material before adsorption. The second sample was prepared by agitating 1 g of AC with 10 mg/l RB and 10 mg/l RR mixture dyes solution (4 h, 200 rpm, and pH=7), then the residue was filtered and dried in oven. The two samples were submitted to the FTIR analyses using FTIR SPECTRALAB device (type: MB 3000, ABB).

#### II.II.II Batch experiments and models

Batch experiments were carried out in 250 ml flasks containing 0.2, 0.4, 0.6, 0.8, and 1 g of AC and 100 ml of each dye solution. These experiments were performed at the same initial concentration of 10 mg/l for each dye. The flasks were placed in a shaker (Edmund Buhler, 7400 Tubingen Shaker-SM 25) with constant shaking speed at 300 rpm for 4 h at room temperature and pH=3. Adsorbent was separated using centrifuge and then filtration. The residual concentration of dye was measured using UV-visible absorption spectrophotometer type: APEL PD- 303

UV JAPAN. Each experiment was repeated three times and the results given were the average value.

The effects of pH, temperature, shaking time, initial dye concentration, and agitation speed on the removal efficiency were studied in a batch reactor. Effect of pH was measured using a 10 mg/l of each dye concentration at room temperature, and the pH range was from 2-10. The temperature effect experiment was conducted using 10 mg/l dye concentration, and the solution temperatures were 10, 20, 30, 40, and 50°C. To study the effect of contact time on the removal efficiency, 11 flasks were used in this experiment. Each flask was filled with 100 ml of 10 mg/l dye solution with 1 g of AC at room temperature and the pH was fixed at 3, the agitation speed was started at 300 rpm and then the samples were taken at the specific time (10, 20, 30, 40, 60, 80, 100, 120, 150, 200, and 240 min.). Effect of initial dye concentrations were conducted using 5, 10, and 20 mg/l dye solutions at room temperature. In order to identify the effect of agitation speed variation on the removal efficiency, beaker of 2 liter is filled with 1 liter of each dye solution and agitation started before adding the 10 g of AC, and then samples were taken for each 1, 2, 5, 10, 15, 20, 30, 45, 60, 90, 120, 150, 180, 240 min to be measured of their remaining concentration. The optimum pH of removal of each solution that obtained from equilibrium isotherm experiments was fixed for each solution before agitation process started at 200, 300, 400, 500 and 600 rpm.

In this study the effect of multisolute interactions on the adsorption capacity onto AC was investigated using binary mixture of dyes. Mixture of RR and RB dyes was subjected to the adsorption process using 10 mg/l of each dye concentration. Six flasks containing 0.5, 0.8, 1, 1.5, 2, and 3 g of AC and 100 ml of mixture dye solution were placed in a shaker with constant shaking speed at 300 rpm for 2 h at room temperature and pH=3. Adsorbent was separated using centrifuge and then filtration. The residual concentration of each dye was measured using UV-visible absorption spectrophotometer.

The uptake is calculated using the following equation:

$$q_e = \frac{(C_o - C_e)V}{m} \quad (1)$$

Also removal efficiency is defined as:

$$\% \text{ Removal} = \frac{C_o - C_e}{C_o} \times 100 \quad (2)$$

#### II.II.III Kinetics models

The dynamics of the adsorption process in terms of the order and the rate can be evaluated using the kinetic adsorption data. A number of kinetic models have been used to describe the adsorption rate in batch operation.

##### Pseudo-first order model

The pseudo-first-order kinetic model has been widely used to predict dye adsorption kinetics. The pseudo-first-order rate expression suggested originally by Lagergren is based on the solid capacity is expressed as follows:

$$\frac{dq_t}{dt} = k_1(q_e - q_t) \quad (3)$$

Where  $q_e$  and  $q_t$  are the amount of solute (mg/g) adsorbed onto the adsorbent at equilibrium, and at time  $t$ , respectively, and  $k_1$  is the rate constant (min<sup>-1</sup>). Integrating

and applying the boundary conditions,  $q_t=0$  at  $t=0$  and  $q_t=q_e$  at  $t=t$ , Eq.(3) takes the following linear form:

$$\ln(q_e - q_t) = \ln q_e - k_1 \cdot t \quad (4)$$

*Pseudo-second order model*

Pseudo second-order adsorption kinetic rate equation is expressed as Eq. (5).

$$\frac{dq_t}{dt} = K_2(q_e - q_t)^2 \quad (5)$$

Integrated of the Eq. (5) for the boundary conditions  $q_t=0$  at  $t=0$  and  $q_t=q_e$  at  $t=t$ , becomes the linear form as Eq. (6):

$$\frac{t}{q_t} = \frac{1}{K_2 q_e^2} + \frac{1}{q_e} t \quad (6)$$

*Intra-particle model*

The possibility of intra-particle diffusion of adsorbate onto the adsorbent by using the intra-particle diffusion model Eq. (7) [11].

$$q_t = K_p t^{1/2} + C \quad (7)$$

$$\text{or, } \ln(q_t - C) = 0.5 K_p \cdot t \quad (8)$$

An intra-particle diffusion rate can be expressed in terms of the square root time. The mathematical dependence of  $q_t$  vs.  $t^{0.5}$  is obtained if the sorption process is considered to be influenced by diffusion in the spherical particles and the convective diffusion in the solution.

The experimental isotherm data were fitted with the two well-known adsorption isotherm models: Langmuir (Eq. (9)); Freundlich (Eq.(10)):

$$\frac{C_e}{q_e} = \frac{1}{q_m K_L} + \frac{C_e}{q_m} \quad (9)$$

$$\text{Log } q_e = \log K_F + 1/n \log C_e \quad (10)$$

The equilibrium model parameters were evaluated and optimized by linear regression using STATISTICA program version 6. The model's fitness was significant as indicated by the coefficient of determination ( $R^2$ ) where a large  $R^2$  value indicates a better fit.

### III. RESULTS AND DISCUSSION

#### III.I Characterization of adsorbent material

Fig.1 shows the results of FTIR spectroscopy analyses of AC before and after adsorption of two dyes. The characteristic absorption bands of hydroxyl and amine groups were identified at 3551 to 3414  $\text{cm}^{-1}$ , C=O of the carboxylic groups or ester groups at 1738, S=O of the sulfonates groups and COO<sup>-</sup> groups of the fatty acids at 1367  $\text{cm}^{-1}$ , the wave number at 1210  $\text{cm}^{-1}$  was attributed to the P-O-C links of the organic phosphate groups [10, 12]. Some bands in the fingerprint regions could be attributed to the phosphate groups [13]. So that, the results of FTIR spectrum showed that the hydroxyl and carboxyl groups could be very effective for capturing these two dyes molecules.

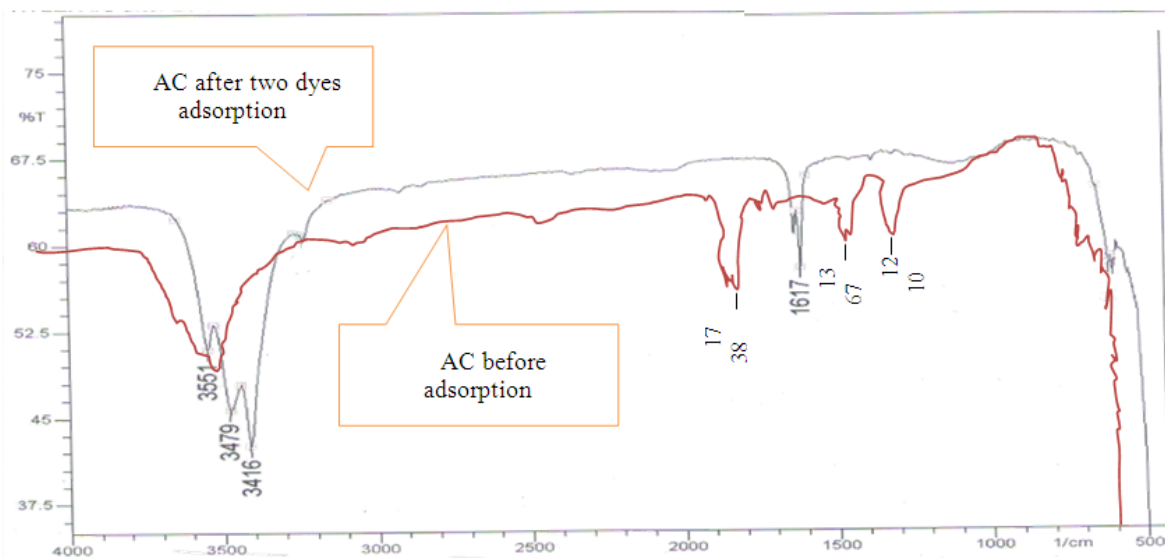


Fig.1. FTIR spectra obtained for AC before adsorption (red), and AC following RB and RR dyes adsorption (gray)

#### III.II Effect of pH

The efficiency of adsorption process is pH dependent due to the variation in pH value leads to variation of surface properties of adsorbent and the degree of ionization [14], therefore, dependency of sorptive removal of dyes onto activated carbon was studied by employing equilibrium adsorption tests as outlined earlier and the experimental results are depicted in Fig.(2a). According to the results that gathered, it has been found that the solution

pH played a significant role in the biosorption process. From this figure, it can be seen that the best pH value for removal were 4 and 3 for RR and RB dyes, respectively. At  $\text{pH} < 3$ , the active surface sites of the adsorbent were dissociated [15], so that the adsorption capacity was decreased at pH value of 2.

Therefore, this pattern of dyes removal is characteristic of anion sorption. At acidic pH, the surface of activated carbon becomes positively charged and this facilitates

sorption of dyes cations. The reason for the reduction of sorption capacity at higher pH values may be explained by the competition between  $\text{OH}^-$  and dye anions. Also, certain chemical compounds in RB and RR dyes such as phenols and acids that are proton donors become anions when the solution pH reaches the equilibrium condition of the compound, in this case the adsorption decreased with increase in pH [16].

### III.III Effect of initial concentration

Fig.(2b) shows the variation of the percentage removal with the variation of dyes concentration from 5 to 20 mg/l at 1 g of AC and pH=3. The results indicated that the percentage removal was not altered greatly from concentrations between 5 to 10 mg/l, this may be due to the amount of AC used in this experiment contains enough adsorption surfaces and sites for this concentration range [17], thereafter the percentage removal rapidly decreased with the further increase in concentration to 20 mg/l. The decrease in the percentage removal of dye can be explained with the fact that all the adsorbents had a limited number of active sites, which have become saturated above a certain concentration [18].

### III.IV Effect of temperature

The effect of temperature on the removal efficiency has been investigated within a temperature range of 15 to 50 °C and the results are depicted in Figure (2c). It can be seen the maximum percentage removal for each of the two dyes occurred between 20-30 °C. Several authors showed that further increases in temperature (above 30 °C) lead to a decrease the percentage removal. This may be attributed to an increase in the relative desorption of the dye from the solid phase to the liquid phase, deactivation of the biosorbent surface, destruction of active sites on the biosorbent surface due to bond disruption [19, 20]), or due to the weakness of the sorbent active site binding forces

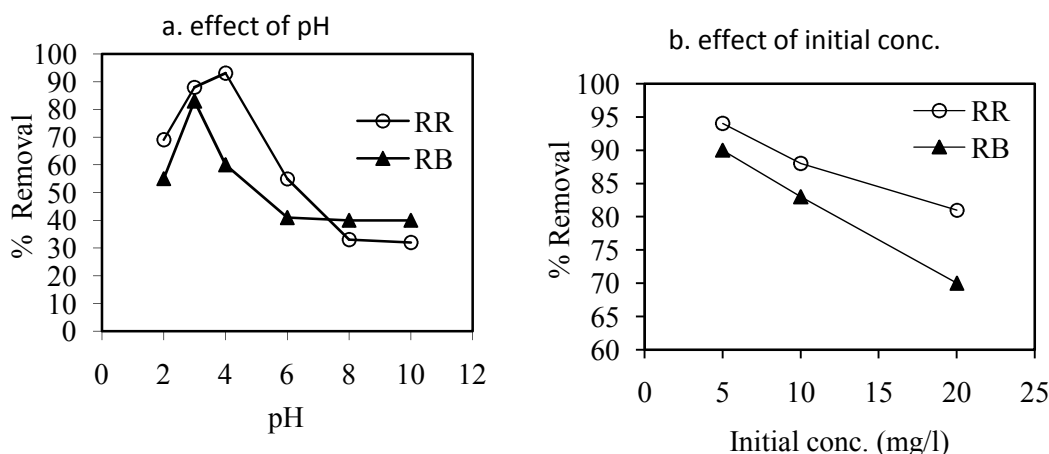
and the sorbate species and also between the adjacent molecules of the sorbed phase [21]. It can be seen from Fig. (2c) that the variation of temperature from 20 to 30 °C has minimal effect on the biosorption process, so that experiments can be carried out at room temperature without adjustment.

### III.V Effect of contact time

It was very important to know the required time to reach equilibrium adsorption. The effect of contact time on the adsorption process was investigated at initial concentration of 10 mg/l and pH=3. The system was subjected to an agitation speed of 300 rpm using rotary shaker. Fig. (4d) shows the results of adsorption efficiency versus contact time for the two dyes solutions. This figure shows a rapid adsorption of two dyes at the initial time and the equilibrium was attained within 120 min. These results indicate that the adsorption process is chemisorption[22].

### III.VI Effect of agitation speed

Fig. 3(a and b) shows the typical concentration decay curves of RR and RB dyes in batch experiments that carried out at different agitation speeds. The optimum agitation speed needed to achieve maximum removal was found to be 300 rpm and for RR and RB dyes. Fig. (3) shows a fast rate for RR dye removal in the first 50 min. compared with RB dye due to high surface binding and mass transfer rate while slow rate was attributed to the interior penetration. Vijayaraghavan and Yun [23] indicated that with appropriate agitation, the mass transfer resistance can be minimized. Additionally, increasing the agitation rate, the diffusion rate of a solute from the bulk liquid to the liquid boundary layer surrounding particles becomes higher due to the enhanced turbulence and the decrease in the thickness of the liquid boundary layer. Under these conditions, the value of the external diffusion coefficient becomes larger.



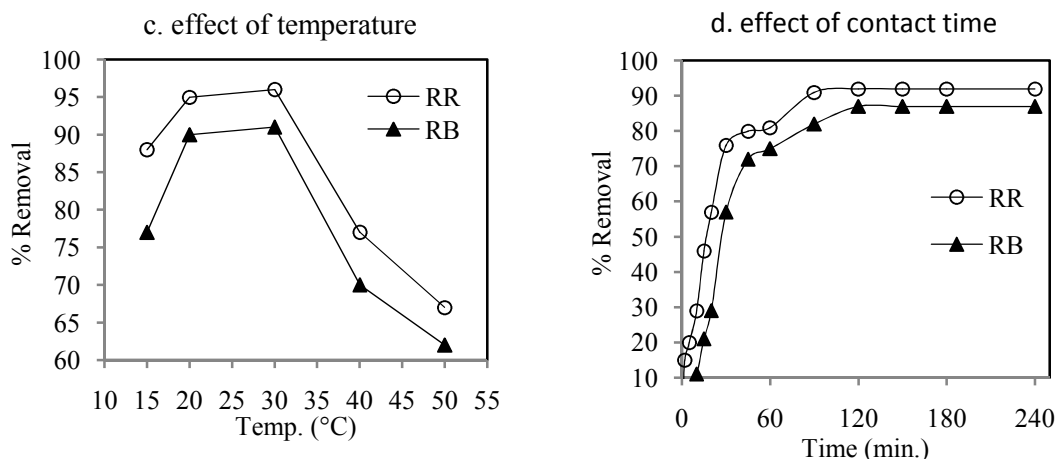


Fig. 2 Effects of physical variables on two dyes adsorption using Activiated carbon

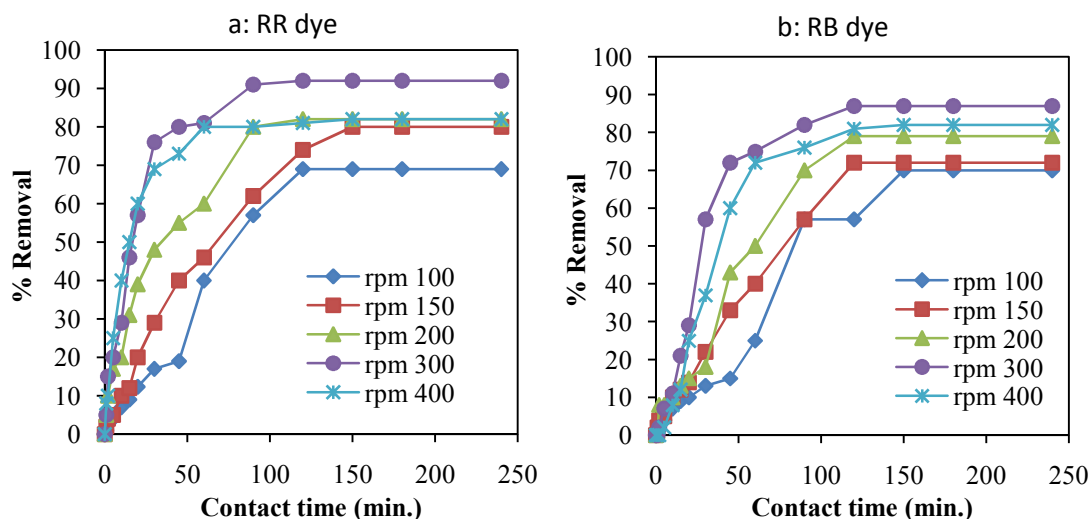


Fig.3. Concentration time decay curves for RR and RB dyes adsorption onto AC at different agitation speed

### III. VII Adsorption Isotherm

Adsorption isotherm are expressed in terms of a relationship between the concentration of adsorbate in the liquid and the amount of adsorbate adsorbed by the unit mass of adsorbent at a constant temperature. Equilibrium adsorption isotherms are useful to understand the sorption interaction as well as to find the adsorption capacity of the adsorbent [24]. In this study, activated carbon is used as an adsorbent for reactive blue and red dyes removal from wastewater. The results showed that the maximum adsorption capacities were 2.15 and 2.89mg/g for RB and RR dyes, respectively.

The determination coefficients of isotherm models are shown in Table (2) for RB and RR dyes adsorption. This table indicates that the Langmuir model provides the best fit as judged by its correlation coefficient for the two dyes. These results indicate the monomolecular adsorption of RB and RR dyes onto activated carbon. The plot of theoretical and experimental equilibrium isotherm for two dyes is presented in Fig. (4). It can be seen from that the isotherm curve is of favorable type.

Fig's.(5 and 6) show the results of single and binary adsorption capacity of RR and RB dyes, respectively. It can be shown that the maximum uptake of two dyes was decreased to 2.89 and 1.33 for RR and RB dyes, respectively. The decrease in adsorption capacity in binary system when compared with the single dye adsorption is due to the competition between dyes molecules for binding sites presented in AC wall and show clearly the interactive effect of the dyes on each other. These results are agreed with Allen et al. [25]. Their results showed that the adsorption capacity of an individual dye decreased in the presence of a second or a third dye. They reported that reduced capacities could be attributed to a combination of a number of factors. These include: (i) interaction between dyes in solution; (ii) change of the adsorbent surface charge due to adsorption; (iii) competitive adsorption between dyes for active sites on the carbon surface where displacement effects replace the other dyes from the adsorption sites.

In single component system the adsorption isotherm curve rises steeply at low liquid phase concentration

indicating complete adsorption with a plateau formed when the maximum adsorption capacity is achieved. However, in multicomponent adsorption, a less regular isotherm shape is found, possibly indicating that the concentration of the dye being adsorbed increased and

then decreased, i.e., a displacement effect. The results showed that the RR dye has a higher affinity for AC than RB dye, as the reduction in RR adsorption uptake was 27% and 38% for RB dye. Also these results indicate a low degree of completion between RR and RB dyes [26].

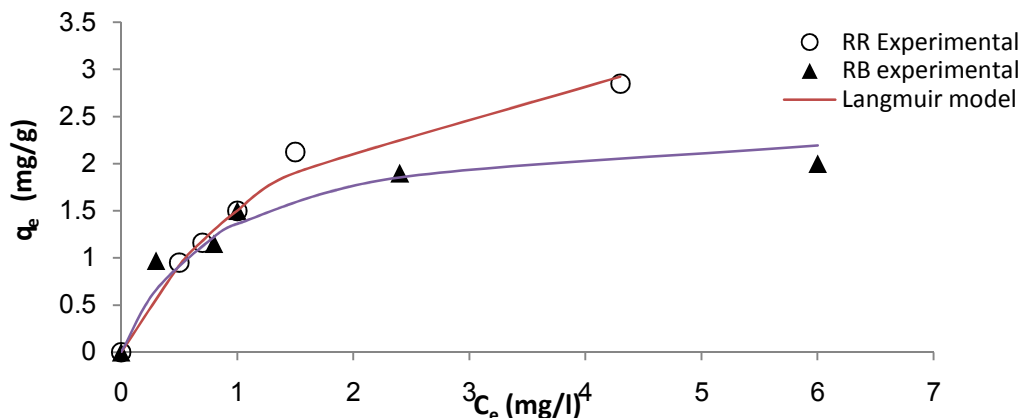


Fig.4. Equilibrium isotherm data for RR and RB dyes (pH=2, room temperature, agitation speed=300 rpm, contact time=2 h.)

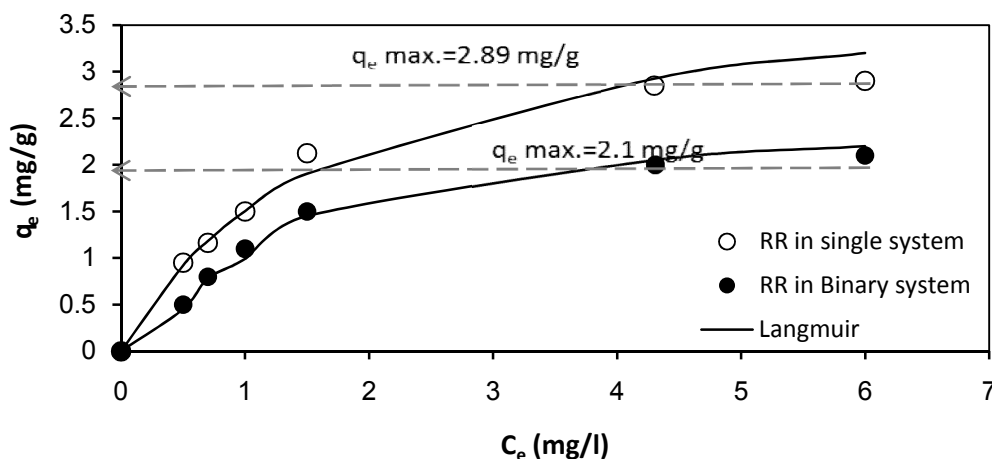


Fig.5. Equilibrium isotherm data for RR dyes in single and binary system (pH=2, room temperature, agitation speed=300 rpm, contact time=2 h.)

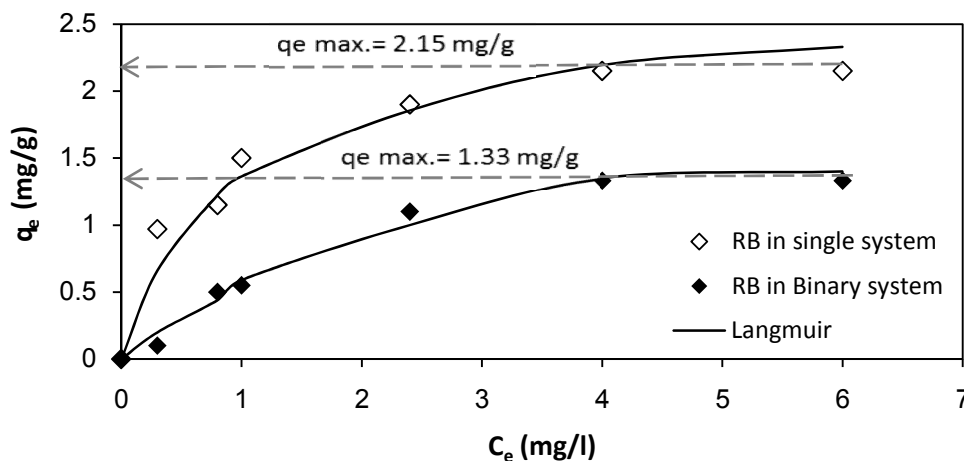


Fig.6. Equilibrium isotherm data for RB dyes in single and binary system (pH=2, room temperature, agitation speed=300 rpm, contact time=2 h.)

Table 2: Langmuir and Freundlich constants

Dye	C <sub>0</sub> ppm	Temp. °C	Langmuir model			Freundlich model		
			q <sub>m</sub> (mg/g)	K <sub>L</sub> (l/mg)	R <sup>2</sup>	K <sub>F</sub> (l/g)	n	R <sup>2</sup>
RB	10	35	2.5	1.28	0.985	1.44	2.77	0.940
RB	10	45	4.08	0.78	0.980	1.69	2.4	0.910
RB	10	55	5	0.56	0.972	1.73	1.83	0.870
RR	10	35	4.1	0.578	0.950	1.44	1.87	0.901
RR	10	45	3.88	0.8	0.860	1.304	1.31	0.823
RR	10	55	4.12	1.14	0.993	1.836	1.50	0.911
RB with RR	10	Room temp.	1.45	1.1	0.909	---	---	---
RB with RR	10	Room temp.	3.01	0.334	0.935	---	---	---

### III.IX Kinetic study

The adsorption data at optimum agitation speed for the two dyes solutes were analyzed in terms of pseudo-first, second order and intra-particle diffusion mechanisms. The rate constant (K<sub>1</sub>) using pseudo-first order rate expression for RB and RR was obtained from the slope of the linear plots of ln(q<sub>e</sub>-q<sub>t</sub>) against t for each solute using Eq.(4). While, the rate constant (K<sub>2</sub>) of pseudo-second order was obtained from the slope and intercept of plots of 1/q<sub>t</sub> against 1/t using Eq.(6). The K<sub>p</sub> of intra-particle model was found from the slope of plotting eq. (8). The values of

the rate constants with the corresponding correlation are presented in Table (3) for both mechanisms and for the two dyes, according to these results, the experimental data for single and binary systems followed Pseudo-second order. It is clear from R<sup>2</sup> values for pseudo-second order model are very high (> 0.877) compared with pseudo-first order and intra-particles diffusion models. These results suggest that this model successfully describes the kinetics of the adsorption of RB and RR onto activated carbon and refers to that the process is chemical sorption [27].

Table 3: Calculated kinetic parameters for Pseudo-first order, Pseudo-second order, and intra-particle diffusion models for RB and RR dyes with correlation coefficients

Dye	Pseudo-first order kinetic model		Pseudo-second order kinetic model		Intra-particle diffusion model	
	K <sub>1</sub> (1/min)	R <sup>2</sup>	K <sub>2</sub> (mg/g min)	R <sup>2</sup>	K <sub>p</sub> (mg/g s <sup>0.5</sup> )	R <sup>2</sup>
RB (single system)	0.0193	0.841	0.0232	0.994	0.123	0.83
RR (single system)	0.019	0.988	0.0165	0.991	0.127	0.91
RB (binary system)	0.007	0.850	0.0187	0.877	0.0475	0.9
RR (binary system)	0.0152	0.801	0.085	0.998	0.054	0.73

## IV. CONCLUSION

The present study evaluates the reactive red and blue dyes removal in single and binary systems by using activated carbon. The results showed that the activated carbon could be used efficiently to remove reactive blue and red dyes from aqueous solution. The maximum percentage removal was 85 and 92 % for reactive blue and red dyes, respectively. The pH hardly changed the sorption capacity in the range 2–6. The variation of temperature from 20 to 30 °C has minimal effect on the biosorption process, so that all the experiments were carried out at room temperature. In the binary system, reactive red dye has a higher affinity for activated carbon than reactive blue dye. Experimental data indicated that competitive adsorption for active sites on the carbon surface results in a reduction in the overall uptake capacity of the reactive dyes investigated. The equilibrium adsorption capacities decreased in binary systems (as compared to single dye systems), the reduction was 27% and 38% for reactive red and blue, respectively. The results indicated that Langmuir model provided the better correlation of experimental data in single and binary systems. The pseudo-second-order rate equation fitted well experimental data of two dyes (better than the pseudo-first-order rate equation).

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## NOMENCLATURE

C	Constant of Intra-particle diffusion model
C <sub>o</sub>	Initial concentration of adsorbate
C <sub>e</sub>	Concentration at equilibrium (mg /l)
K <sub>F</sub>	Freundlich constant for adsorbate (l/mg)
K <sub>1</sub>	The rate constant of pseudo first order adsorption (min <sup>-1</sup> )
K <sub>2</sub>	The rate constant of the pseudo second order adsorption (g.mg <sup>-1</sup> . min <sup>-1</sup> )
K <sub>L</sub>	Coefficient of Langmuir model (l/g)
K <sub>p</sub>	The intra-particle diffusion rate constant(mg.g <sup>-1</sup> s <sup>-1/2</sup> )
q <sub>e</sub>	Adsorption uptake (mg/g)
q <sub>m</sub>	Maximum adsorption uptake (mg/g)
q <sub>t</sub>	Adsorption uptake at time t,(mg/g)
n	Freundlich Constant